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William J. Rodgers
Lecturer, University of Ulster, Newtownabbey, Co. Antrim, Northern Ireland

Banihan Gunay
Lecturer, University of Ulster, Newtownabbey, Co. Antrim, Northern Ireland

Alan Woodside
Professor of Highway Engineering, University of Ulster, Newtownabbey, Co. Antrim, Northern Ireland

ice
Institution of Civil Engineers

Rheological and electrical properties of modified bitumen

W. J. Rodgers BEng, PhD, B. Gunay MSc, PhD, CMIHT, CMILT and A. Woodside MPhil, CEng, FICE, FIEI, FIAT, FIQ, MIHT, MCIWEM

A pavement heating system was investigated as an alternative to chemical application on highway pavements. As a result, surface course asphalt mixes needed to be modified to develop improved electrical properties. The bituminous binder was outlined as that part of asphalt mixes that could be modified with filler-characterised additives. These additives included carbon black, pulverised fuel ash and iron powder. Samples were prepared and subjected to electrical tests in order to obtain the optimal additive content. The materials were subjected to rheological and electrical performance tests. Pulverised fuel ash had limited influence on the inherent electrical properties, although higher levels of substitution performed better than unmodified bitumen. The carbon black and iron powder were found to improve the capacitive and resistive properties of bitumen. Carbon black was found, however, to give the best predictability for in-service use for asphalt mixes with varying filler-to-bitumen ratios

NOTATION

A	area of the electrodes (cm^2)
C	capacitance of the material (pF)
E	applied voltage (kV)
f	frequency of applied voltage (MHz)
l	distance between the electrodes (cm)
$\boldsymbol{P}$	power input (W)
R	volumetric resistance of the sample (ohm)
$\tan\delta$	loss factor
ε'	dielectric constant
ρ	resistivity (ohm-cm)

1. INTRODUCTION

The present research is part of a wider project investigating dielectric heating as a possible alternative to de-icing chemicals. These chemicals melt ice and snow on pavement surfaces during the winter as a result of exothermic and endothermic reactions. Poor electrical conductors can be heated by dielectric heating. The curing of resins in the timber industry (Pound, 1973) and dehydration of food products (Jones, 1985) has been achieved with dielectric heating. Insulative or dielectric materials are differentiated from conductive materials based on resistivity (Accorsi and Bhatt, 2008). It is known that bitumen is a dielectric material (Whiteoak, 1990). The energy that can be dissipated as heat is defined as the product of the square of an applied alternating voltage, the frequency of the signal, the capacitance of the material, and the dielectric constant of the material (BEDA, 1957). The binder of bituminous mixtures was chosen for modification to suit dielectric heating requirements.

The binder phase of a bituminous mix is made from bitumen and filler. Limestone is a popular filler in asphalt mixes. This can, however, be modified with different fillers. Modifiers have been used to improve the performance of bituminous binders and asphalt mixes. The types of modifiers include carbonaceous materials, polymers and other filler-type materials. Carbon black is often derived from pyrolytic processes using tyres (Chaala *et al*., 1996) and softwood bark charcoal (Chebil *et al*., 2000). Polymers incorporated into polymer-modified binders (PMB) are ethylene vinyl acetate (EVA; Gonzalez *et al*., 2004), polybutyl acrylate (PBA), polyethyl acrylate (PEA), polymethyl acrylate (PMA), styrene butadiene styrene (SBS; Airey *et al*., 2008) and polyvinyl chloride (PVC; Singh *et al*., 2003). Materials from various sources such as rice husk ash (Saxena *et al*., 1984), hydrated lime (Lesueur and Little, 1999), fly ash (Asi and Assa'ad, 2005), pulverised fuel ash (pfa; Nicholls *et al*., 1998) and cement and fly ash (Oikonomou, 2003) have also been assessed.

Previous studies investigating modified binders focused on rheological performance (Chebil *et al*., 2000). The present study, however, looks at the suitability of modified binders as a charge carrier for asphalt mixes heated by radio-frequency heating. An earlier study looking at the electrical properties of asphalt used carbon black as a substitute for limestone filler (Ahmedzade and Geckil, 2007) and the results showed that carbon black improved both electrical and mechanical performance. The present study includes three modifiers from different sources.

The aim of the present study was to investigate the modification of the bitumen–filler matrix, incorporating three different filler materials with petroleum bitumen. These modified mortars were investigated to compare the electrical properties with pure bitumen. The influence of the modifiers on rheology was also measured using classical tests. Figure 1 represents the dispersion of filler in bitumen as the level of addition increases. Figure 1(a) shows unmodified bitumen, whereas Figures 1(b) to (d) illustrate the addition of increasing amounts of filler. As the amount of filler increases, it is enveloped by the bitumen.

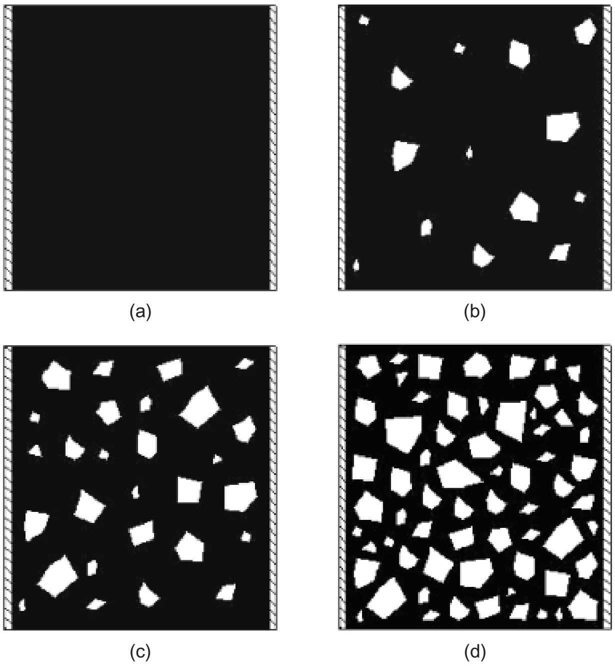

Figure 1. Filler dispersion in bitumen

2. EXPERIMENTAL

2.1. Materials

Three modifying additives were chosen, specifically focusing on the substitution of the limestone filler that is traditionally used in asphalt mixes. The additives chosen were pfa, carbon black and iron powder. The pfa was sourced locally from a coal-fired power station. The carbon black is a commercially available material and is used as a doping material in plastics to encourage electrostatic discharge. The iron powder is also a commercially available product and is traditionally used in manufacturing for powder metallurgy processes. The chemical composition and physical analysis is presented in Table 1.

2.2. Blending of modified bituminous binders

The pfa, carbon black and iron powder additives were dried for a period of 4 h at 110°C. Previous studies used particles that were all less than 45 μm to provide the best homogeneous modified bitumen (Chebil *et al.*, 2000). However, for this work particles passing a 63 μm sieve were used in accordance with the specification for filler materials specified in BS 4987–1: 2005 for constituent materials of coated macadams (BSI, 2005). Blending of the materials was carried out at a temperature of 140–150°C. The modifiers were added to the bitumen in small quantities of approximately 0·5 teaspoon. This was done to facilitate smooth mixing and to avoid agglomeration of the modifiers. The additives were introduced to the bitumen as a function of the volume. This was done to facilitate the large differences in material densities. The materials were added at five levels, namely 14, 27, 36, 41 and 57% vol. for bitumen. These levels of substitution were based on actual filler contents of dense bitumen macadams (DBM) and stone mastic asphalt (SMA) mixes. One grade of bitumen was used for this study. The penetration grade of the bitumen was 125 pen.

2.3. Softening point and penetrability sample preparation and method

For the classical rheological tests, samples were produced in accordance with BS EN 1426: 2000 for the needle penetration of bitumen and bituminous binders (BSI, 2000a) and BS EN 1427: 2000 for the softening point of bitumen and bituminous binders (BSI, 2000b). The first stage in sample preparation required the modified binders to be heated at 160°C and stirred as described in Section 2.2 to ensure a representative uniform sample of the material. For the softening point tests, the modified binders were poured into testing rings and cooled at 5°C. The samples were then placed in ball-centring guides in water and stored for 24 h. Testing was carried out using a magnetic hot plate with a magnetic stirrer to ensure uniform heating. Instead of using a thermometer to measure temperature, a thermocouple was used and coupled with a datalogger. This was done to stop

Chemical	Symbol	pfa	Carbon black	Iron powder
Oxygen	O	–	–	0·15
Carbon	C	5	100	0·05
Iron	Fe	–	–	99·8
Chlorine	Cl	0·013	–	–
Calcium oxide	CaO	4·15	–	–
Sodium oxide	Na_2O	0·35	–	–
Silicon dioxide	SiO_2	52·2	–	–
Titanium dioxide	TiO_2	1·5	–	–
Sulfur trioxide	SO_3	0·8	–	–
Iron oxide	Fe_2O_3	3·95	–	–
Magnesium oxide	MgO	0·38	–	–
Potassium oxide	K_2O	0·93	–	–
Aluminium oxide	Al_2O_3	28·5	–	–
Physical analysis				
Percentage passing 63 μm		100	100	100
Percentage passing 45 μm		–	99·99	2
Particle density: kg/m^3		2060	264	7800

Table 1. Chemical composition and physical analysis

anomalous heating. The penetration tests required heating and stirring of the modified binder prior to pouring into test containers. After a sample of the material had been poured, the material was cooled as quickly as viably possible as problems due to fines settling out of mastics during testing at high temperatures have been reported (Grabowski and Wilanowicz, 2008). This was found to be the case, particularly with the iron powder and was attributed to the high density of the material.

The sampling rate used for the softening point tests was two samples of each modified binder that adhered to the repeatability precision for modified bitumen as outlined in BS EN 1427: 2000 (BSI, 2000b). For the penetration tests, the sampling rate was four valid determinations per modified sample. The maximum difference between the highest and lowest determinations was 0·4 mm for bitumen with a penetration grade between 50 and 149 and 0·6 mm for bitumen with a penetration grade between 150 and 249.

2.4. Tests samples for electrical tests

BS 7737 – 1: 1995 was used as a basis for the production of samples for electrical tests (BSI, 1995). The standard discusses the physical characteristics of the samples that were considered when producing the samples for this work. The physical appearance of a sample is given in Figure 2. The retaining surround of the sample was made from medium density fibreboard (MDF). Other materials such as high-density polyethylene (HDPE), high-density polystyrene (HDPS) and acrylic were evaluated. These materials were found to deflect as a result of the pouring temperature of the modified binder. The MDF performed best in this respect. The material chosen for the electrodes was aluminium. It was cost-effective and easy to cut to shape in comparison with copper and stainless steel. The electrodes were visually inspected to ensure surfaces were free from blemishes that could affect the bitumen–electrode interfacial bond. The thickness of the MDF board was 6·35 mm (0·25 in.) material and the aluminium sheet was 0·8 mm thick. The cut-outs in the MDF were 250 mm × 250 mm ± 1·0 mm. The tolerance on the cut-outs was to allow for the tolerance (250 mm × 250 mm ± 0·5 mm) and the alignment of the electrodes is shown in Figure 3. The samples that were used to determine electrical properties of materials needed to be symmetrical about the three world axes as shown in Figure 4.

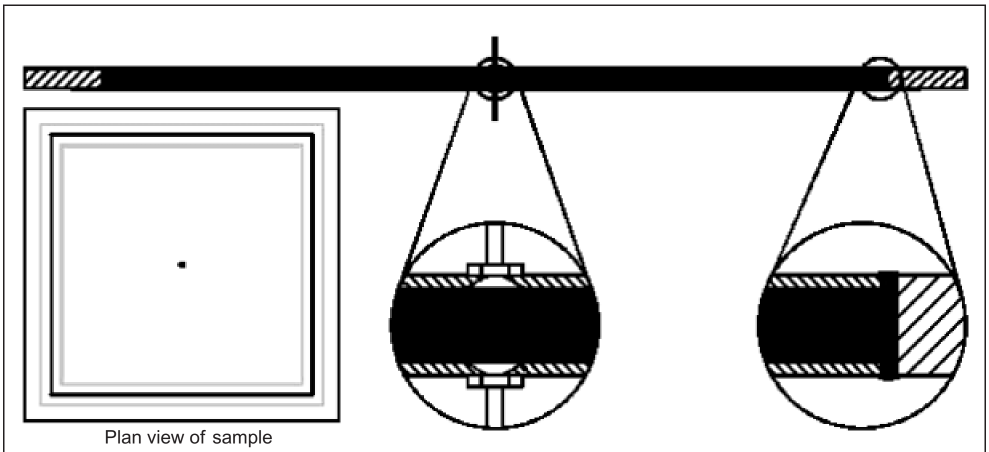

Figure 2. A test sample

2.5. Test procedure for electrical properties – sample preparation and method

Tests for electrical properties were carried out on an AIM LCR Databridge 401. This piece of equipment was sufficient as it allowed low capacitance to be measured with a precision of 1 picofarad (pf) and high resistance to be measured in the range of ohms (Ω) to mega-ohms (MΩ). Materials could be tested at two frequencies: 100 Hz and 1 kHz. Each sample was placed in turn into a specially made holder. Standard 50 Ω electrical connectors were used to connect the sample and the equipment. Each reading was taken after 1 min once the reading stabilised as a result of fluctuations that occurred during connection and alignment. The databridge is based on a piece of equipment called a Wheatstone bridge that uses the values of known resistors to determine that of an unknown resistor or a dielectric material in the form used here, as shown in Figure 5.

3. RESULTS AND DISCUSSION

3.1. Softening point and penetrability

The effect of the modifying materials on softening point as a function of modifier concentration is presented in Figure 6. It can be seen that the softening point increased with increasing quantities of the modifiers. Figure 6 shows that the increase in softening point was linear with the addition of the modifiers. However, carbon black exhibited better correlation with an exponential trend. This relationship is similar to that observed by Chaala *et al.* (1996) who reported a much more abrupt increase in softening point. It has also been shown that an exponential relationship between modifier concentration and softening point exists (Chebil *et al.*, 2000).

Carbon black had the most noticeable effect in comparison with the pfa and iron powder. During the ring and ball tests, the sample that contained 41 and 52 vol.% exhibited a different form of softening. There was much more deformation of the sample and the ball penetrated through the test sample, detaching much of the material from the ring. This has been previously observed (Chaala *et al.*, 1996) and it was proposed that these high levels of modifiers formed hard gels with the bitumen. This was further explained in terms of the chemical make-up (maltenes and asphaltenes) and bonds within the bitumen and that the bitumen could become more fluid as a result of high maltene oil content due to greater bonding between modifier particles and asphaltenes.

The effect of the modifiers on penetrability is presented in Figures 7 to 9. All three materials caused a reduction in penetrability which led to increased stiffness. The pfa and iron powder both had similar effects of penetration with increasing concentration. Carbon black was different, particularly at 30°C at which there was a larger reduction in

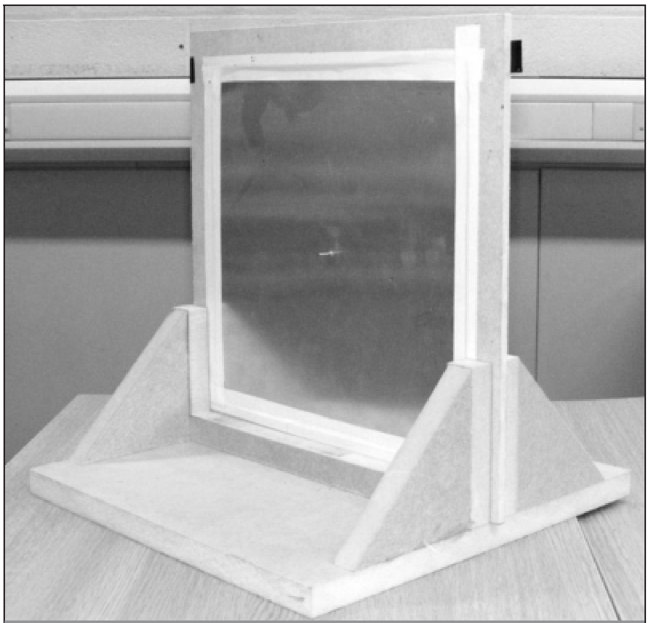

Figure 3. View of sample demonstrating pick-up points and electrode alignment (not to scale)

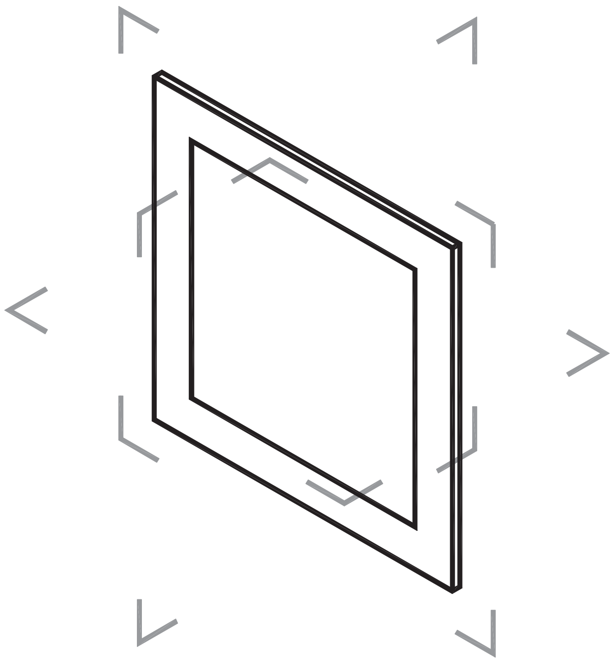

Figure 4. Check for symmetricity

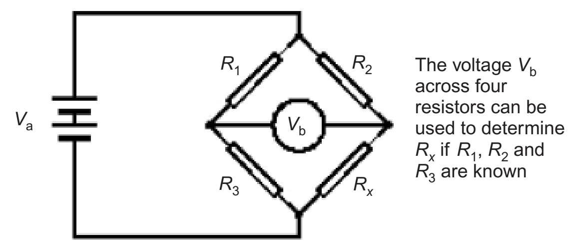

Figure 5. Wheatstone bridge

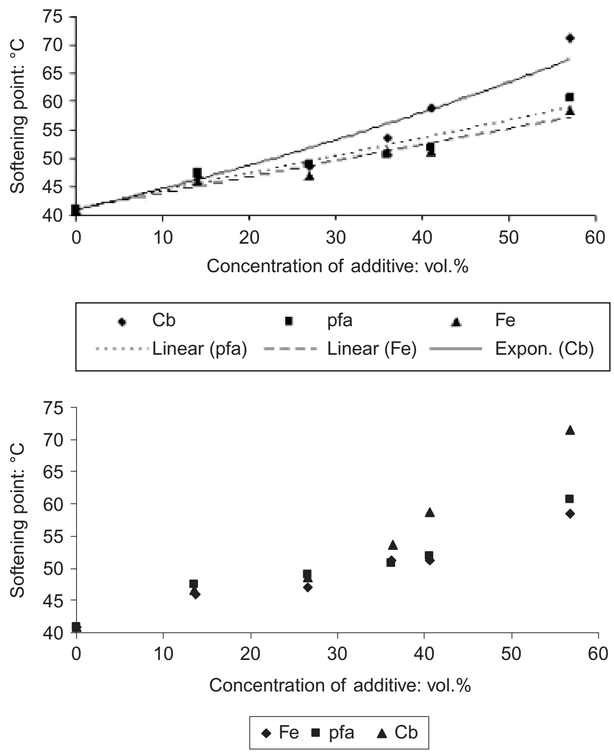

Figure 6. Softening point of modified binders

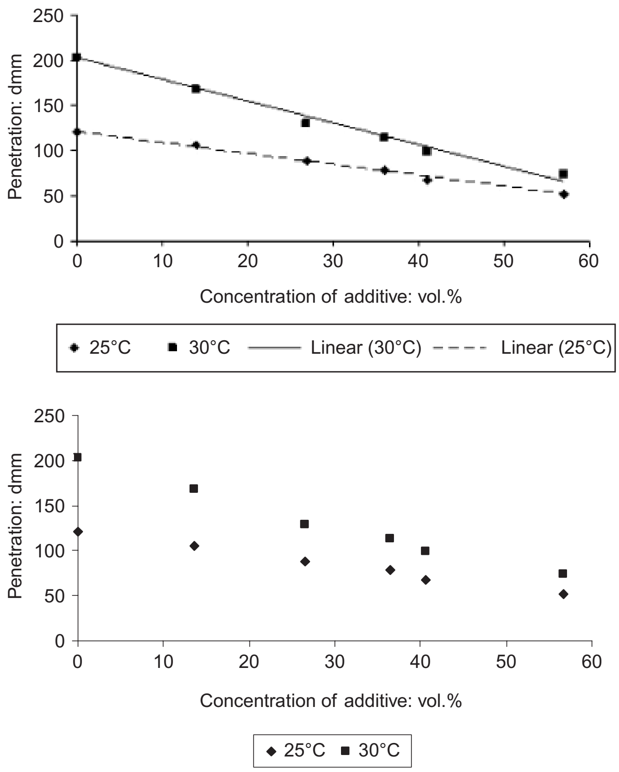

Figure 7. Penetrability of modified binder containing carbon black

penetrability. This reduction in penetrability has been explained as the enhancement of viscosity caused by the creation of new bonds between filler particles and bitumen asphaltenes (Chebil *et al.*, 2000) and so higher viscosity is related to decreased penetration and greater stiffness. Similar

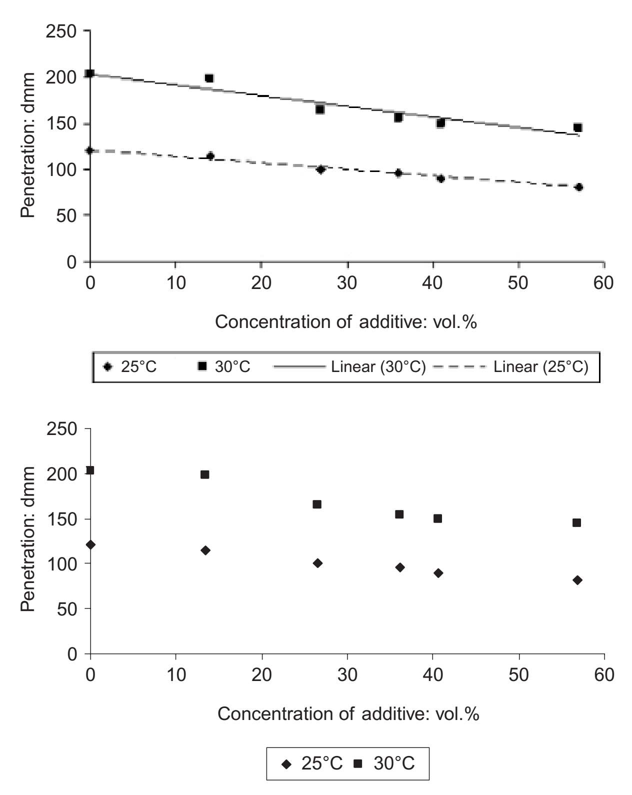

Figure 8. Penetrability of modified binder containing pfa

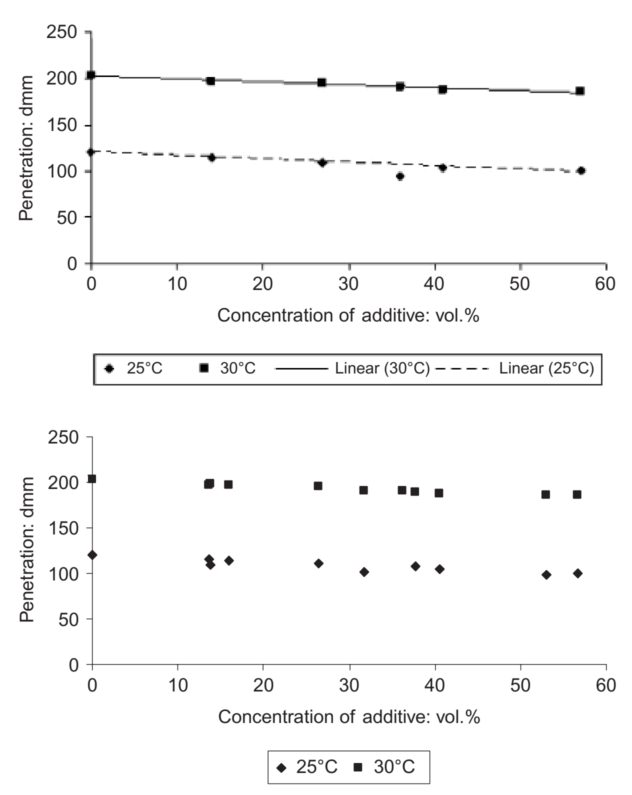

Figure 9. Penetrability of modified binder containing iron powder

results have been observed with the use of carbon black (Chaala *et al.*, 1996). The slopes presented relate to thermal susceptibility. Therefore, the larger the gradient of the slope, the lower the thermal resistance of the modified binder. Of the three additives evaluated, only carbon black had any influence on the thermal susceptibility. The effect on penetrability was in accordance with softening point data.

3.2. Electrical capacitance and dielectric constant of modified binders

The effect of the additives on capacitance at 100 Hz and 1 kHz are presented in Figures 10 and 11. It can be seen that frequency had no effect on the capacitive properties of the modified binders. Capacitance increased with increasing concentration of additive for both carbon black and iron powder, with iron powder showing the best increase. The results for pfa as a modifier would suggest that there is little advantage in using pfa for dielectric applications, and that the inclusion is harder to predict. Carbon black and pfa followed a linear trend offering good correlation. This was true of iron powder, although a better correlation was shown with an

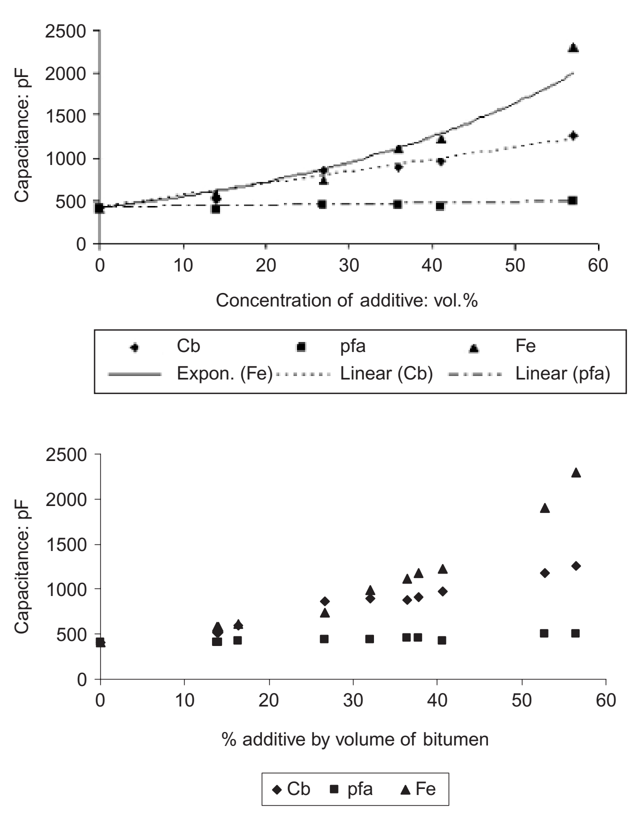

Figure 10. Capacitance of modified binders at 100 Hz

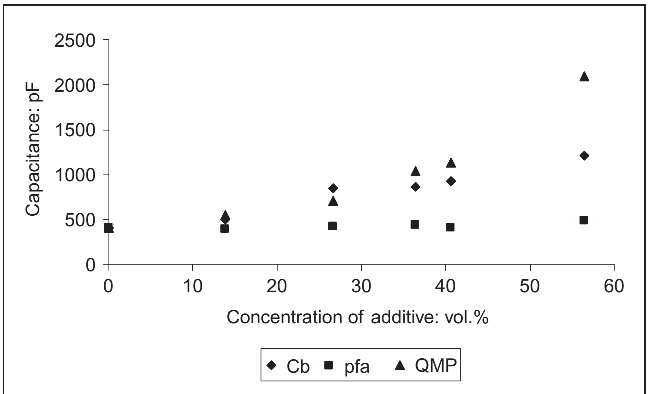

Figure 11. Capacitance of modified binders at 1 kHz

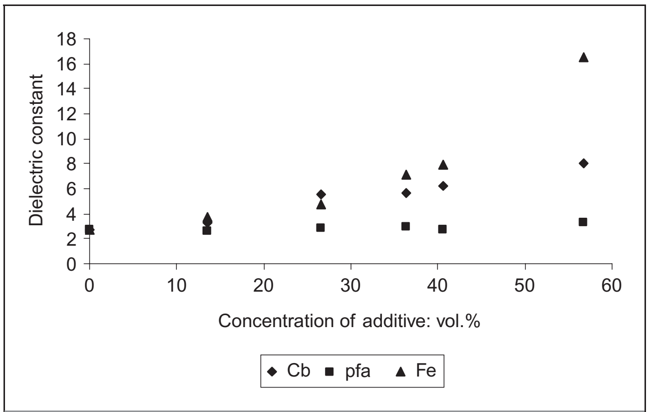

Figure 12. Dielectric of modified binders

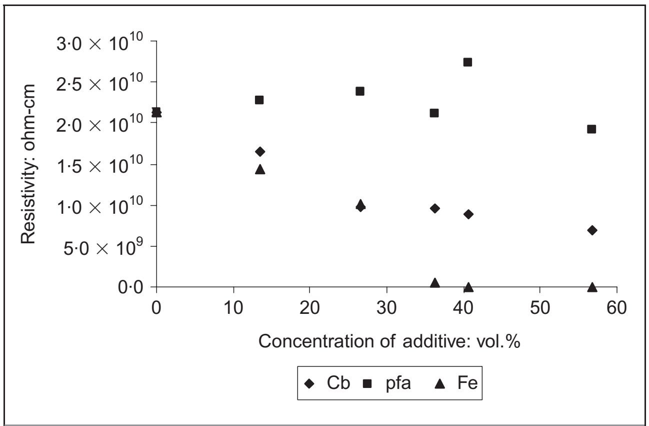

Figure 13. Resistivity of modified binders at 100 Hz

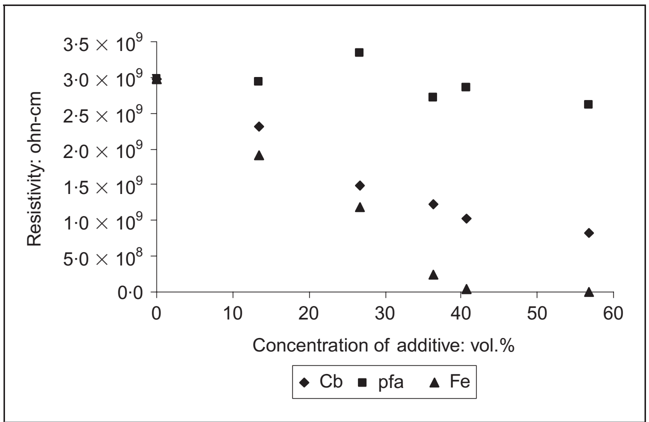

Figure 14. Resistivity of modified binders at 1 kHz

exponential plot. Varying the levels of modifiers shows that no material exhibits a threshold as can be found when examining the resistivity of modified binders.

It has been stated that the amount of power that can be dissipated in a dielectric material is a function of the frequency of an input voltage, the capacitive quality of a dielectric material, the dielectric constant of that material and the loss factor as shown in Equation 1 (Matsuoka and Wilson, 1986). This relationship shows that the higher the capacitance of a dielectric material, the more power can be dissipated in that material. Therefore, as capacitance increases with increasing addition of the modifiers, the more suitable are these materials for improving the performance of bituminous mixtures in dielectric heating systems.

$$P = 2\pi f E^2 C \varepsilon' \tan\delta \quad (1)$$

The dielectric constants for the modified binders are presented in Figure 12. The dielectric constant is based on the ratio of the capacitance of a capacitor with a dielectric material and a capacitor with an air gap. The higher dielectric constant exhibited by the modified bitumens, particularly at higher levels of addition, satisfy the relationship with respect to the power that can be dissipated in dielectric materials (Matsuoka and Wilson, 1986).

3.3. Electrical resistivity of modified binders

Figures 13 and 14 show the resistivity of bitumen filled with carbon black, pfa and iron powder. The three modifiers exhibited different effects on the virgin bitumen, namely by making the bitumen more insulative, more dissipative and more conductive. The inclusion of pfa yielded conflicting results at the two test frequencies. When tested at 100 Hz, pfa exhibited an increase in resistivity. At 1 kHz, however, there was a decrease in resitivity that was similar to the other modifiers. The increase in resistivity means that the pfa was causing the bitumen to be more insulative. Carbon black caused the resistivity to reduce in a linear fashion. This finding differs from those of a previous study (Cui *et al.*, 2007) in which it was shown that as the quantity of carbon black increased (for several different blends) the resistivity passed through different phases of reduction. It has been shown that the rate of drop of resistivity can decrease at different rates (Cui *et al.*, 2007). The points of these changes are referred to as percolation thresholds.

The percolation threshold is a level of additive modification defined as parts per hundred of resin (phr), which is similar in definition to the vol.% (percentage by volume of bitumen). The data presented here show that there was no percolation threshold for carbon black-modified bitumen and that the reduction in resistivity at higher levels of substitution could be predicted more accurately than for pfa and iron powder.

Iron powder showed a point at which the rate of resistivity decrease changed from rapid to slow. This occurred at 36 vol.%. Although the rate of decrease of the iron powder could be addressed as a linear decrease, better correlation of data was achieved using a polynomial. However, the polynomial regression did not satisfy the projected resistivity of iron powder at 100%. The percolation threshold of 36% needs to be further investigated as this level of substitution should yield two distinct linear relationships for the iron powder. Previous research has indicated that the decrease in resistivity occurs as a result of contact or apparent contact between filler particles, thereby allowing electron hopping or tunnelling to occur (Cui *et al.*, 2007). This means that when filler particles are covered in a bitumen film, as the film becomes thinner the electric charge can be transferred between particles through the bitumen film.

The resistivity data can be used to define the modified materials. Figure 15 shows how materials can be classified that high levels of filler place greater demand on bitumen as a result of the increased surface area of filler. This means that the bitumen forms thin films around the filler particles and thus the volume of bitumen deceases. This reduces the effect of the bitumen on the volumetric resistance of the sample. The difference in performance between the modifiers tested then depends upon the resistivty of these materials. Lack of studies regarding the resistivity of pfa means a representative value for resistivty was difficult to define, but the chemical composition of pfa shows that it is made up of chemicals that contain oxygen and yield high resistance to electric current, such as aluminium oxide ($2{\cdot}5 \times 10^6$ ohm-cm) and silicon dioxide (1×10^{15} ohm-cm). By contrast, carbon and iron have low resistance of 0·006 ohm-cm and 0·0000089 ohm-cm, respectively, and so the resistivity of bitumen can be reduced, although the choice of filler is important in carrying out this function.

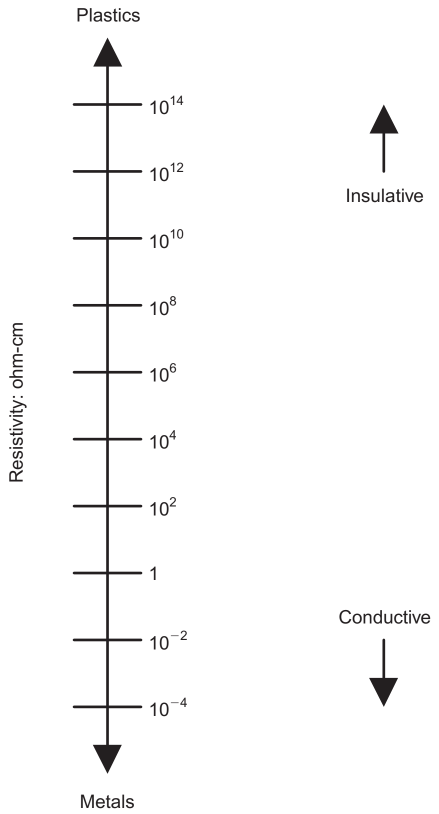

Figure 15. Resistive classification of materials: source: Petrie (2008)

from being insulative to conductive, based on their resistive characteristics. The resistivity of the bitumen modified with pfa remained high in the region of 10^{10} ohm-cm at 100 Hz and 10^9 ohm-cm at 1 kHz and showed that this material was dissipative. The effect of carbon black was similar showing resistivity reducing to 10^9 ohm-cm at 100 Hz and 10^8 ohm-cm at 1 kHz. Again the material remained dissipative. This is advantageous in a material that is to be used for dielectric heating in which it is to dissipate electric power as heat. However, the iron powder showed that at the percolation threshold of 36 vol.% the modified bitumen changed from being dissipative to conductive. Making a material conductive negates its suitability for use in a dielectric heating system. High levels of conductive fillers can cause short-circuit effects that can lead to a conductive nature (Wu *et al.*, 2005). The modified bitumens used were related to bitumen/filler ratios of various asphalt mixtures. Filler choice will depend upon filler content as a function of bitumen content.

The resistivity data presented are based on the volumetric resistance of the samples tested according to Equation 2

$$\rho = \frac{RA}{l} \tag{2}$$

When the unmodified bitumen was tested, the material within the electrodes was uniform and homogeneous. As the addition of the modifying additives increased however, the resistivity changed, based on the properties of the component materials within the modified bitumen. It has already been established

4. CONCLUSIONS

All three fillers used had similar effects on bitumen rheology. Softening point and penetrability tests were used. The softening point increased and the penetrability decreased with increasing levels of modifier. Carbon black had the most noticeable effect, exhibiting the largest increase in softening point. Penetration data suggested that the temperature susceptibility decreased particularly in modified binders containing carbon black. The capacitance of bitumen containing three additives was evaluated with the capacitance being measured at 100 Hz and 1 kHz. There was no significant change in capacitance at the two frequencies tested. These data were verified by deriving the dielectric constants for the modified binders which were constant. Both carbon black and pfa demonstrated purely linear relationships with increasing quantity. However, the iron powder showed better correlation of data when observing the exponential relationship. Iron powder and carbon black both exhibited characteristics that are beneficial to dielectric heating.

The electrical resistivity of the modified binders was tested. Tests were carried out at 100 Hz and 1 kHz. It was shown that the effectiveness of pfa for dielectric heating applications was greatly impacted by results which suggested that resistivity can be reduced or increased to a more insulative nature. The carbon black and iron powder showed that resistivity decreased with increasing addition of modifiers. Carbon black showed a linear reduction in resitivity whereas iron powder showed a polynomial relationship that suggests a maximum substitution of iron powder at 36 vol.%. The reduction in resistivity shown suggests these materials would perform well in dielectric heating for asphalt applications. Further research needs to address the use of these modifiers on pavement behaviour and performance.

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